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ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMA LARGE CALIBER WEAPON SYSTEMS LABORATORY DOVER, NEW JERSEY

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The interior ballistics problem associated with the erratic and abnormal burning of gun propellant grains is addressed in terms of the mechanical behavior of the propellant.

Compressive mechanical properties, grain failure, and burning performance of two triple base propellants tested as a strain rate of 1000% per second and at ambient temperatures ranging from 20°C to -60°C are presented.

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20. ABSTRACT (cont)

As the test temperature is lowered the propellants undergo a change from ductile to brittle mechanical stress-strain behavior; the associated grain failure proceeds with a decrease in the amount of plastic nonfragmenting type deformation and an increase in the extent of fragmenting type fracture. The extent of fracture or breakup changes the prescribed surface area of the characteristics of the propellant design. Closed bomb tests with normal and with mechanically-failed grains show increases in relative quickness sufficient to produce unsafe gun firing conditions.

Changes in the mechanical properties of nitroguanidine propellants observed over a period of 1 year suggest that aging can cause propellant embrittlement.

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INTRODUCTION

The objective of this work was to see how the mechanical properties of propellant grains could relate to the erratic and unsafe functioning of multiple-grain gun propellant charges.

Continuing efforts to understand the causes of abnormal propellant performance occurring during the ignition and combustion phases of the interior ballistic cycle have implicated grain fracture as a potential mechanism for abnormal burning (refs 1 through 5). The prevailing hypothesis is that propellant grains in various stages of burning are accelerated and are impacted against the confines of the gun chamber. This produces grain fracture that can result in sudden and significant increased burning surfaces and increased gas evolution (pressurization) rates. This hypothesis formed the basis for this work and accordingly localized the test conditions of temperature and strain rate used to those associated with the military use of propellants, and further to those most contributing to grain breakup. The loading conditions employed are temperatures at or above the lower limit of terrestial temperatures of military interest, approximately -60°C, and a rate having times to failure of 2 to 10 milliseconds, and consistent with rise times observed in pressurization curves of gun firing records (ref 6).

The emphases in this program were to identify loading conditions which could produce brittle propellant grain behavior, to illustrate the effects of such brittleness on grain failure and on propellant burning and performance, and to suggest parameters indicative of the brittleness and mechanical breakup to be expected of the propellant grains during gun firing.

EXPERIMENTAL

Mechanical Tests

The fast rate testing was performed in a pneumatic/hydraulic type loading system developed for this installation in 1961 by Hesse-Eastern. A sketch of the apparatus is shown in figure 1 and its operation is as follows. The system is pressurized and maintained in a "ready" state by an equilibration of pressures above (N_2 gas) and below (H_2 0 liquid) the movable crosshead-linked piston, the puncturing of a polyester diaphragm suddenly vents the restraining pressurized liquid and allows the pressurized gas to expand rapidly, driving the movable crosshead toward the stationary crosshead; spacers are used to stop the moving crosshead at various distances from the stationary crosshead. A strain rate of approximately 10 second⁻¹, or a loading rate with millisecond rise times, hereafter referred to as the fast rate, is obtained.

Test temperatures for the fast rate were obtained using Optron temperature regulating equipment. A temperature chamber was designed to fit within the confines of the load column (fig. 2a); the temperature variation during a l hour sample conditioning was \pm 3°C.

The load and the change in crosshead separation at the fast strain rate were measured using a load cell and a differential transformer (LVDT), respectively. The LVDT coils were mounted on the stationary crosshead and the LVDT core on the movable crosshead. A photograph of the load column showing the LVDT and the temperature chamber is given in figure 2b. The transducers signals versus time were inputted to a dual beam scope and photographed. Typical load-time (lower) and displacement-time (upper) traces are shown in figure 3.

Data reduction was performed in a film reader system that magnified the oscilloscope photographs, and used an x-y cross wire setup for reading and digitizing the traces. This data was then inputted into a computer program to produce stress-strain data and plots.

Closed Bomb Tests

Closed bomb tests are used to assure that the burning and pressure-time characteristics of successive propellant compare with an established standard of the same formulation (MIL-STD-286B, Method 801.1).

The bomb is instrumented to record the rate of pressure buildup versus pressure. Comparison of the test propellant data with that of the standard provides relative performance information from measurements of the relative quickness and relative force.

Thermal Analysis

Measurements of linear expansion coefficients and heat capacities for glass transition studies were made using the Dupont 900 Thermal Analysis System with the 940 Thermomechanical Analyzer (TMA) and Differential Scanning Calorimeter (DSC) cell base modules, respectively; the coolant used was liquid nitrogen.

The TMA measurements were made on samples 0.635 cm long and 0.635 cm in diameter. The y-axis sensitivity was 6.5×10^{-4} cm probe displacement per cm of chart, and the heating rate used was 0.2° C per minute.

The DSC measurements were made on chopped or granulated samples. The heat flow rate range was 5 millicalories per second, and the scan rate used was 1.25 K per minute.

Propellants Studied

The compositions of the triple base propellants studied are given in table 1. These propellants are essentially two-phase composites consisting of a crystalline monopropellant, of either nitroguanidine or RDX (cyclonite),

dispersed in a plastic monopropellant binder of approximately a 1:1 ratio of nitroglycerin plasticizer and nitrocellulose polymer.

The cylindrically-shaped propellant grains were solvent extruded. The nitroguanidine base propellant grains were "glazed" and multiperforated and were approximately 2.86 cm long with a diameter of 1.27 cm. Test samples were machined to 1.27 cm lengths for a length-to-diameter ratio of 1, with ends flat and parallel and as normal to the grain axis as possible; the grains were warped. The RDX base propellant was an experimental formulation produced here; it was unglazed and unperforated. Test samples made from strands were machined to 1.27 cm lengths and 1.27 cm diameters with the ends flat and parallel, and normal to the grain axis.

The ends of each propellant sample were coated with graphite at the start of the test. The sample was positioned within the fast rate machine load column, conditioned for approximately I hour at the test temperature, and tested in fast rate compression.

RESULTS AND DISCUSSION

The effect of different ambient temperatures on the fast rate mechanical behavior of the propellants is seen in the corresponding changes in their stress-strain properties. In general, the elastic moduli and the compressive strengths increase as the temperature decreases and the strains to fracture decrease. The stress-strain curves for the nitroguanidine and RDX base propellants are shown in figures 4 and 5. Photographs of the deformed and fractured samples are presented in figures 6 and 7. Deformation at room temperature shows obvious plastic, barrelling-type failure. Exaggerated compression at fast rate and room temperature of the RDX propellant to 50%, and of the nitroguanidine propellant to 80% of their original lengths (deformation times of approximately 50 and 80 milliseconds, respectively) showed grain cohesion with distortion, large fissures, and unpropagated surface cracks.

These data identify the stress-strain curves with the actual mechanical failure exhibited by the propellant samples. Changes in the fast rate mechanical failure of the propellants, from nonfragmenting to fragmenting, occur at approximately -15°C for the nitroguanidine base propellant and -30°C for the RDX base propellant formulation; corresponding strains at fracture are less than two percent. It is important to note that these transition temperatures are meaningful only at this fast strain rate. These data show that these propellants in thermal equilibrium at temperatures less than approximately -30°C deformed at a rate of 10 second (time to fracture - 4 milliseconds) will exhibit multiple-breakup and significantly change the propellant burning designs. Faster strain rates at -30°C or lower temperatures at a strain rate 10 second will result in greater grain breakup and greater changes in propellant burning characteristics.

The effect of the relative amounts of propellant breakup, produced in the fast rate compression tests at temperatures of 20° C and at -45° C, on the burning

characteristics of the propellant grains was examined using the instrumented closed bomb technique. Undeformed propellant grains were burned in the closed bomb to serve as standards for comparison and illustrate the rate of pressure buildup, dP/dt versus pressure, P curve typically expected. techniques have been employed successfully for screening triple base gun propellant lots for safe and unsafe ballistic use (ref 2). Increases in relative quickness greater than 15% were classified and shown to be unsafe. The closed bomb dP/dt-P curves for the nitroguanidine and the RDX base propellant are shown in figures 8 and 9. These show increases in the relative quickness, considerably greater than the 15% used for unsafe propellant lot classification, and changes In the pressure at which the maximum rate of pressure buildup occurs; no significant change in the peak pressure was observed. It is implicit here that the corresponding mechanical behavior and grain failure produced at temperatures above -45°C would result in lesser changes in burning characteristics and that those produced below -45°C should result in greater changes in burning characteristics.

It is necessary at this point to mention that aging may be an important factor affecting the propellant properties in such a way that brittle mechanical behavior and breakup occur under less severe loading conditions of temperature and strain rate. Table 2 lists increases in compressive strength and corresponding changes in the strain-at-fracture observed after a 1 year period in samples of the same propellant lots. It must be noted, however, that the samples were not stored under controlled conditions during this period, and even though this effect was also independently reported by another investigator (ref 7), more systematic and careful studies are necessary to substantiate these incidental observations.

The temperature at which the amorphousness of a polymer or a polymer system changes from a more flexible, rubbery state to a more rigid, glassy state is called the glass transition temperature (identifiable as a ductile-to-brittle transition temperature). During the transition, significant changes in thermal expansion coefficient, viscosity, elastic modulus, etc., can be measured. The addition of a plasticizer, generally, a low molecular weight organic material, to the polymer suppresses the temperature at which the transition takes place. The effect of differing amounts of such a material on the glass transition temperature of nitrocellulose is reproduced in figure 10; note that the plasticizer is tricresyl phosphate and not nitroglycerin.

If a plasticized polymer system, such as the nitroglycerin-nitrocellulose propellant system is chemically and physically stable, and is insensitive to conditions that could alter the plasticizer concentration, then the glass transition temperature could be the most important characteristic parameter of the system. The mechanical properties for the system could then be evaluated in terms of plasticizer content (ref 8), typically illustrated in figure 11, and the generation of similar data at strain rates associated with the interior ballistics cycle would make the mechanical behavior of the propellant more predictable.

Thermal mechanical analysis was performed on both the RDX and the nitroguanidine base propellant to determine if this technique could be used to measure glass transition temperatures in these propellants. The glass transition temperature, Tg, might then be used as an index of the propellant's stability and provide a means of following aging and degradation processes. It could then be useful in predicting premature brittle failure under otherwise routinely safe loading conditions. A curve of the axial expansion as a function of temperature for the RDX propellant is presented in figure 12 showing the glass transition temperature for the RDX base propellant at approximately -57°C. A glass transition is suggested in the same temperature range for the nitroguanidine base propellant, but was not as obvious. The use of differential scanning calorimetry to measure heat capacity and determine glass transition temperatures essentially confirmed the above, that is, a change in heat capacity for the RDX base propellant was found at approximately -57°C, but was not apparent for the nitroguanidine base propellant.

These glass transition temperatures, at -57° C, correlate with temperatures at which the fast rate mechanical breakups of these propellants exhibit advanced degrees of brittleness, figures 6 and 7; the failures occur within the Hookean portions of their stress-strain curves, figures 4 and 5.

These results are preliminary but suggest that the use of glass transition measurements in propellant characterization can have useful merit.

CONCLUSIONS

- 1. Nitroguanidine and RDX base gun propellant grains exhibit brittle stress-strain behavior and fragmentation-type grain failure at ambient temperatures of -15° C and -30° C, respectively, when deformed at a strain rate of 10 inches/inch/ second.
- 2. The effect of fragmentation-type grain failure on closed bomb, dP/dt versus P, curves show:
 - Ballistically unsafe increases in relative quickness.
- b. Shifting of the maximum dP/dt to lower pressures, particularly for the multi-perforated grains (burning changing from progressive to degressive).
 - c. Negligible change in the maximum pressure developed.
- 3. Changes in propellant mechanical properties, such as increases in strength and decreases in strain-to-failure, observed at the end of a l year period, suggest a time-dependent embrittlement for the nitroguanidine propellant. Although the effect of aging requires systematic verification, the implication that resulting brittle propellant behavior and fragmentation-type grain failure would occur under less severe loading conditions or conditions initially regarded to be normal and safe should be considered.

- 4. The glass transition temperatures for the nitroguanidine and the RDX propellants tested were observed at approximately -57°C; these temperatures, coincidentally, correspond to those producing linear brittle stress-strain behavior at the fast rate.
- 5. Glass transition temperature measurements may be useful for characterizing propellant behavior and grain failure, as well as monitoring time-dependent changes taking place within the propellant.

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Table 1. Gun propellants studied

Composition in weight percent

	<u>M30A2</u>	N6260
Nitrocellulose (% nitrogen)	27.40 (12.57)	29.3 (12.0)
Nitroglycerin	22.52	22.7
Nitroguanidine	45.77	5.0
RDX (Class E)	~-	36.5
Ethyl centralite	1.48	1.5
Dioctyl phthalate	~-	5.0
Potassium nitrate	2.83	
Graphite	0.12	

Table 2. Aging effects noted in the fast rate mechanical properties of nitroguanidine propellant tested at the beginning and end of a l year period*

Propellant	Temperature	Mechanical properties	June 1977	June 1978
RAD 69711	Room temperature	Compressive strength (MPa) Strain-at-failure (%)	53.8 5.3	86.9 2.4
	-45°C	Compressive strength (MPa) Strain-at-failure (%)	202.2 6.6	248.4 0.5
RAD 69713	Room temperature	Compressive strength (MPa) Strain-at-failure (%)	62.1 4.9	87.6 2.0
	-45°C	Compressive strength (MPa) Strain-at-failure (%)	211.8 4.4	218.7 0.9

^{*}Data verification needed under more controlled conditions

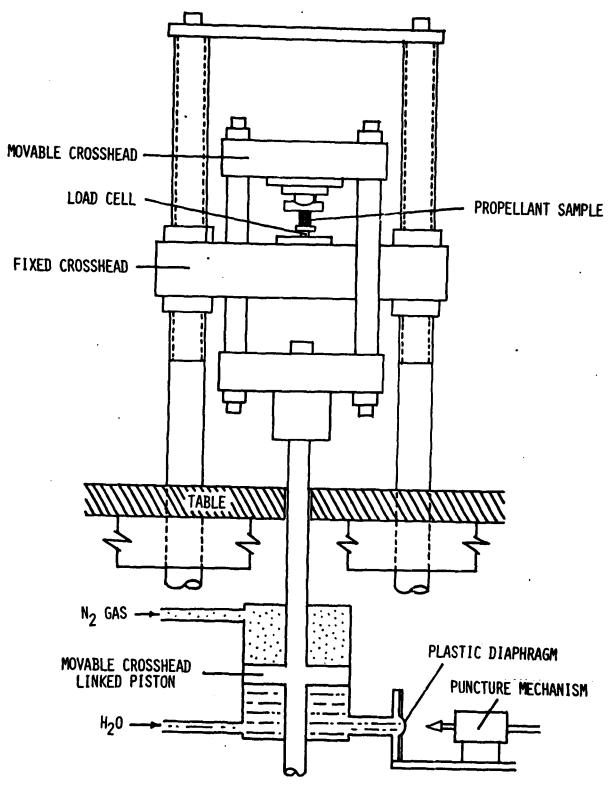
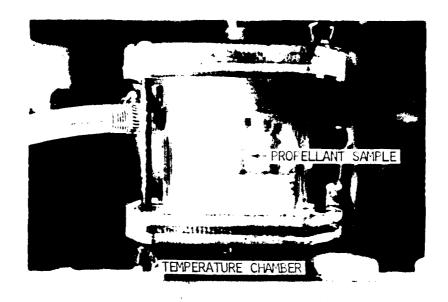


Figure 1. Sketch of the fast rate loading apparatus



(a) temperature chamber

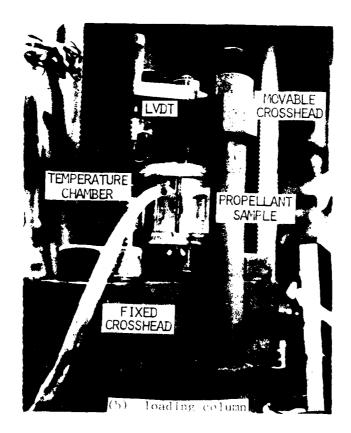
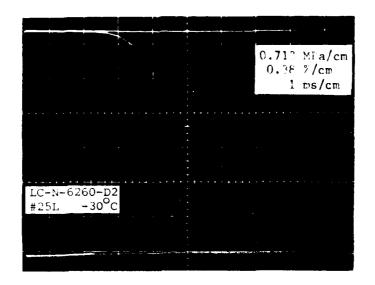


Figure 2. Fast load rate system features



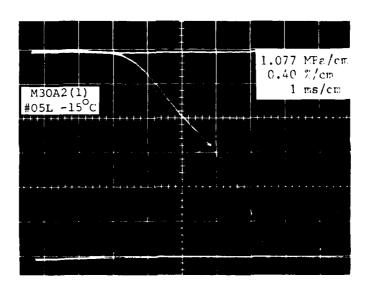


Figure 3. Typical oscilloscope records of fast rate mechanical testing of gun propellant samples (lower trace - load, upper trace - deflection)

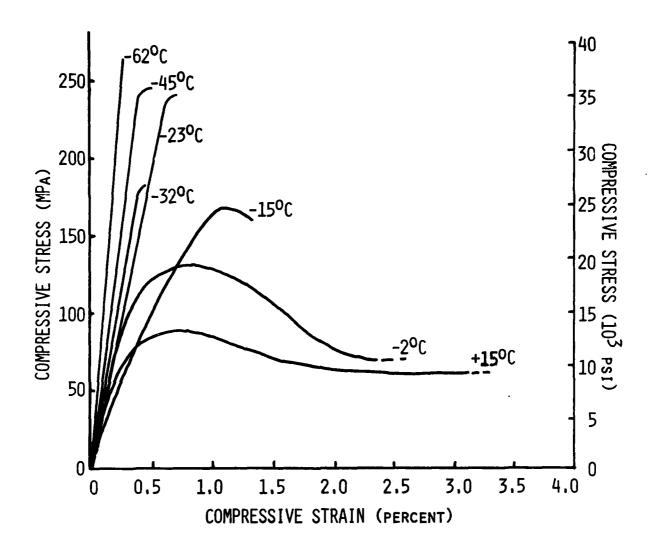


Figure 4. Effect of temperature on nitroguanidine propellant stress-strain curves (times to failure between 5 to 3 milliseconds)

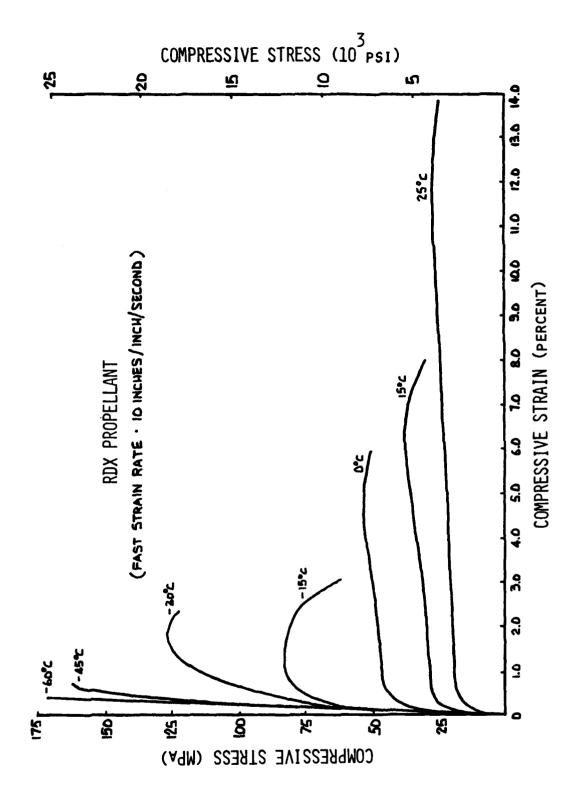


Figure 5. Effect of temperature on RDX propellant stress-strain curves

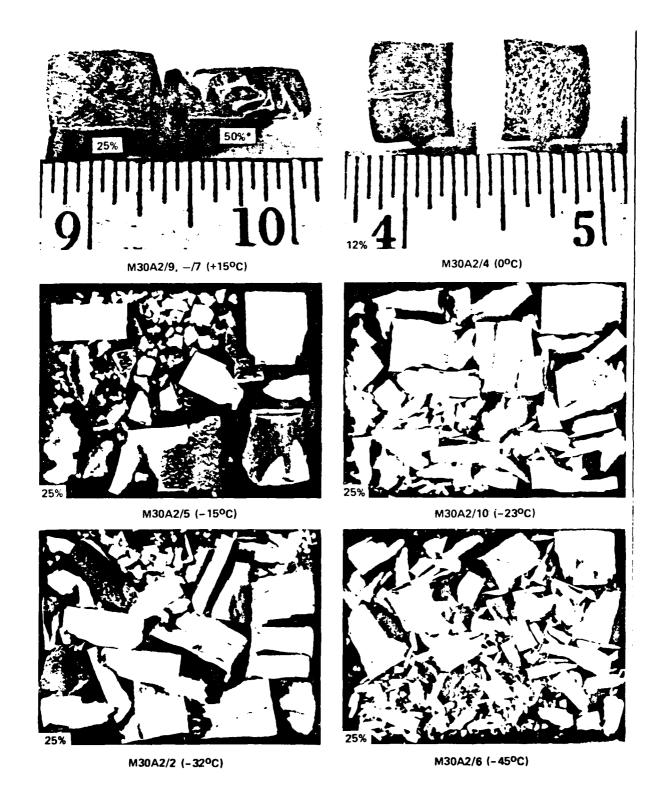
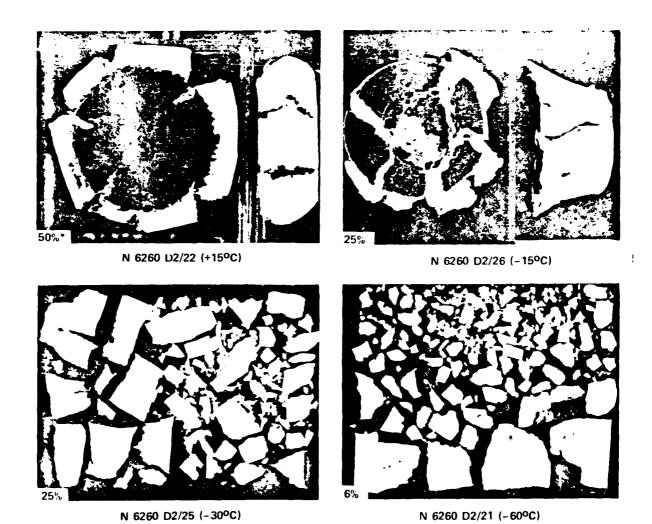


Figure 6. Fast rate compressive failure features of nitroguanidine propellant samples



* APPROX CROSSHEAD TRAVEL IN PERCENT OF SPECIMEN GAGE LENGTH

Figure 7. Fast rate compressive failure features of RDX propellant samples

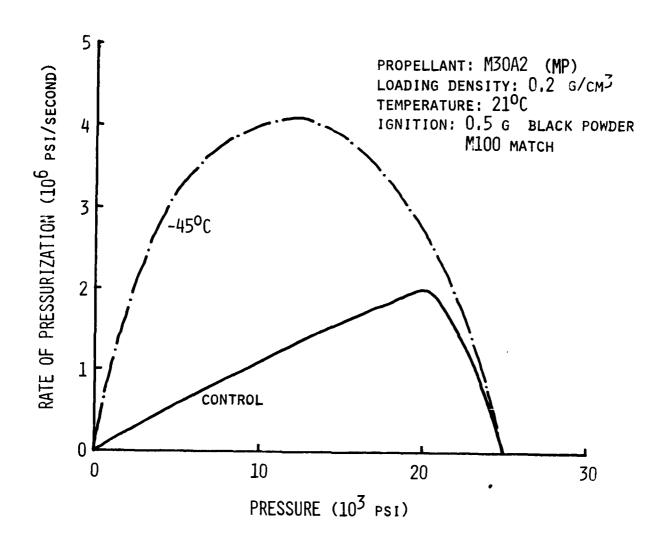
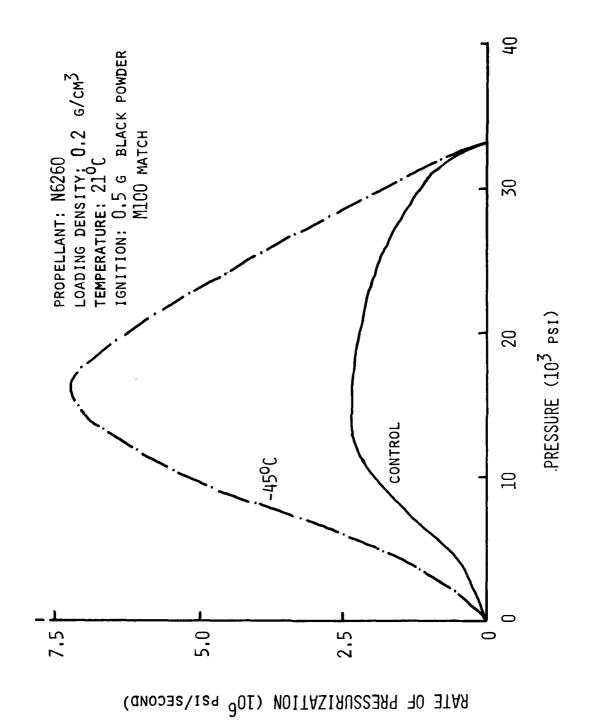


Figure 8. Closed bomb traces of typical (undeformed) nitroguanidine propellant grains and of mechanical-failed nitroguanidine propellant samples (failure produced at -45°C at the fast rate)



Closed bomb traces of typical (undeformed) RDX propellant and of mechanically-failed RDX propellant samples (failure produced at -45° C and at the fast rate) Figure 9.

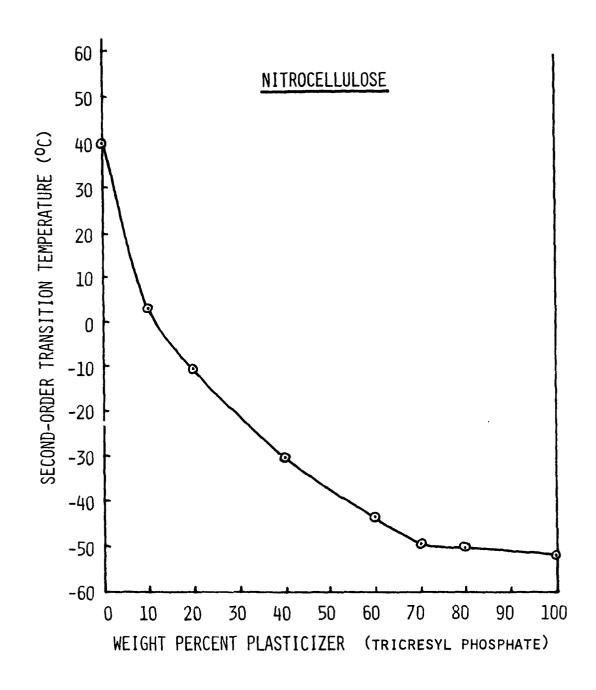
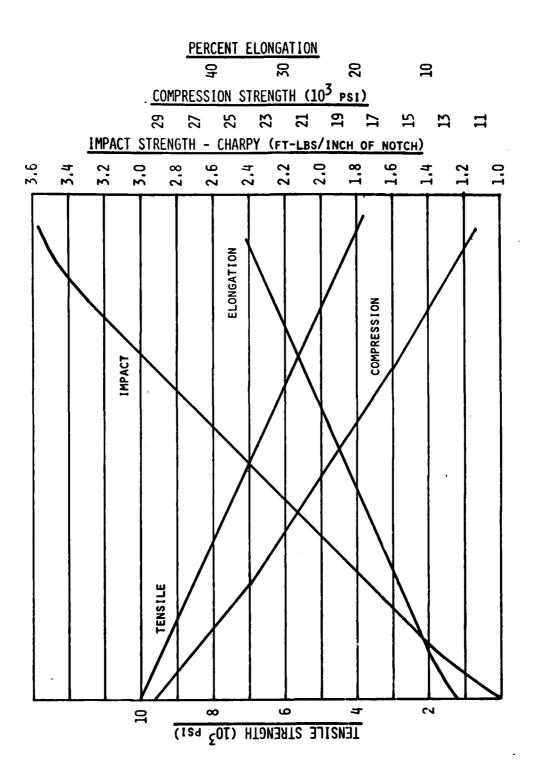
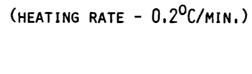


Figure 10. Effect of weight percent of plasticizer on the glass transition temperature of the tricresyl phosphate-nitrocellulose plasticized polymer system



Effect of plasticizer content on physical properties of cellulose acetate Figure 11.

INCREASING PROPORTION OF PLASTICIZER



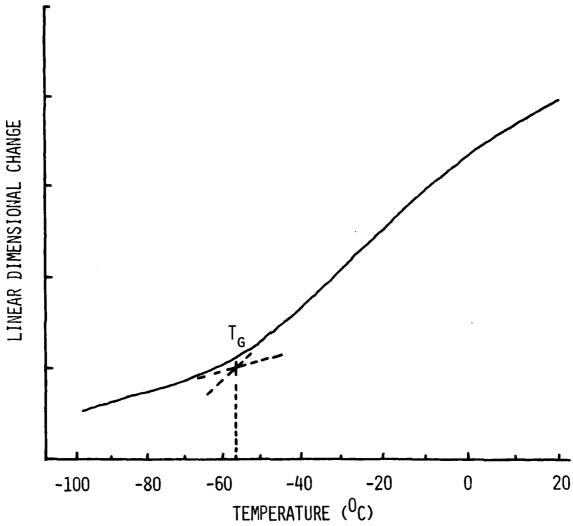


Figure 12. Glass transition temperature determination of the RDX propellant formulation using thermomechanical (TMA) analysis

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